

5187-11-10-134

Integrated Thermal Structures & Materials Overview

Dr. Brian Jensen
NASA Langley Research Center
(757) 864-4271
b.j.jensen@larc.nasa.gov

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

♦ **Resins for transfer molding or infusion processing**

• **POC:**

- Paul M. Hergenrother
- (757) 864-4270
- p.m.hergenrother@larc.nasa.gov

♦ **Nonautoclave processable adhesives**

• **POC:**

- Dr. Brian J. Jensen
- (757) 864-4271
- b.j.jensen@larc.nasa.gov

♦ **Automated Tape Placement Device with e-beam cure**

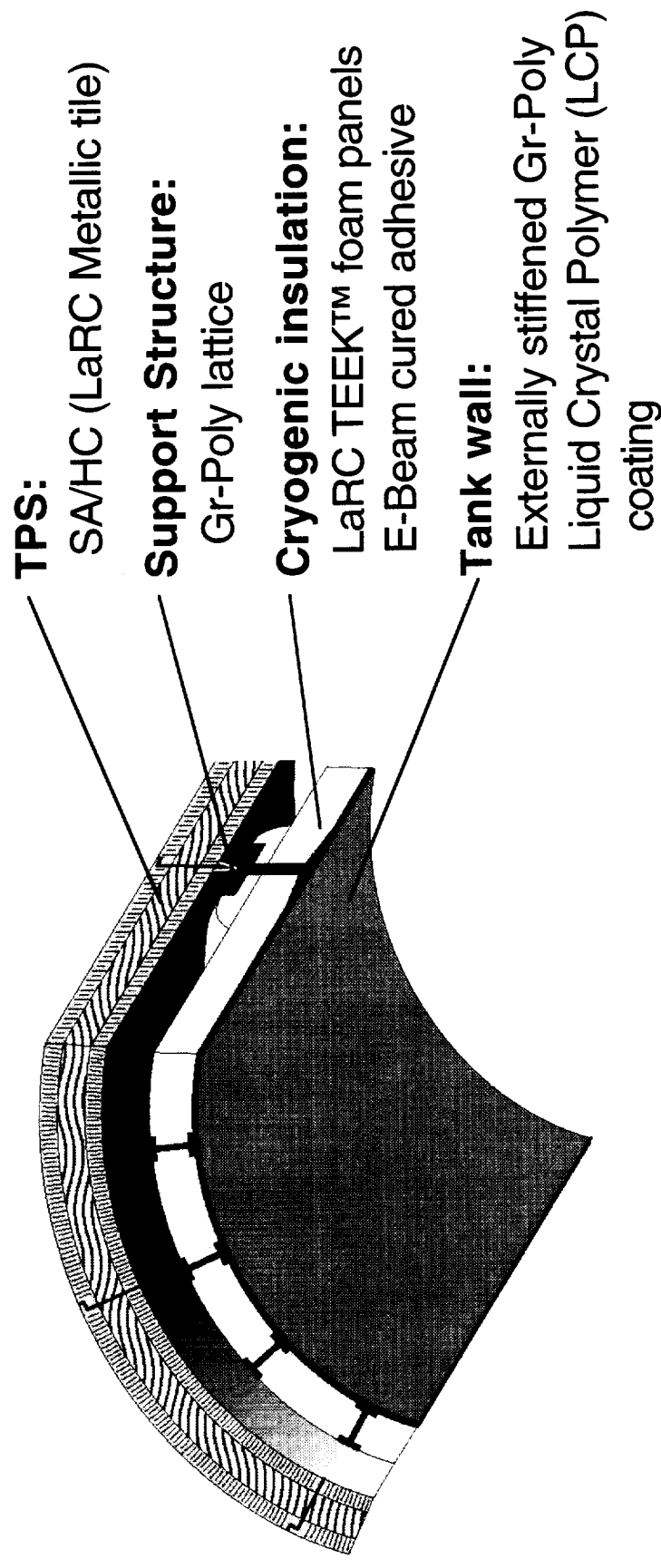
• **POC:**

- Harry L. Belvin
- (757) 864-9436
- h.l.belvin@larc.nasa.gov

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

High Temperature RLV Tank Concept



3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

- ♦ Resins for transfer molding or infusion processing
 - POC - Paul M. Hergenrother, NASA LaRC
- ♦ Nonautoclave processable adhesives
 - POC - Brian J. Jensen, NASA LaRC
- ♦ Automated Tape Placement Device with e-beam cure
 - POC - Harry L. Belvin, NASA LaRC

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

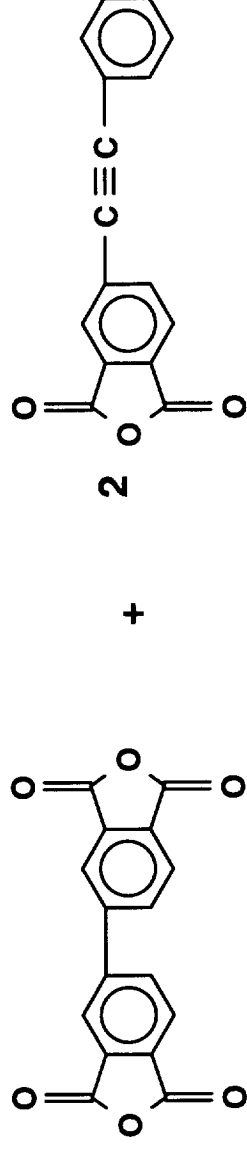
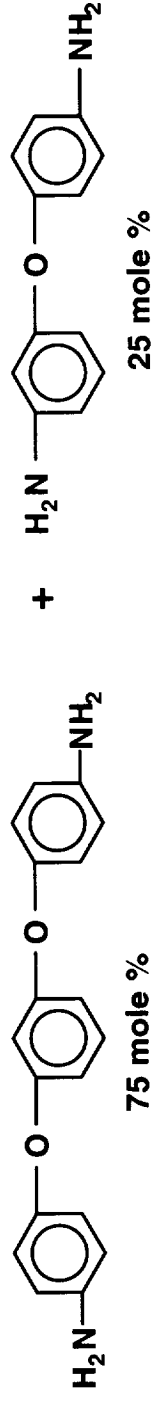
Accomplishments, RTM/RI Resins

- ♦ LaRC prepared 5 resins with Tgs as high as 625°F, <1% volatiles, moderate toughness and low melt viscosity and sent to Boeing or Lockheed Martin
- ♦ GRC prepared 4 resins with Tgs as high as 700°F, <10% volatiles and low melt viscosity and sent to Boeing
- ♦ Boeing successfully fabricated 2' x 2' x 36 ply composites by resin infusion (RI) of stitched preforms from all NASA supplied resins
- ♦ Lockheed Martin successfully fabricated 13" x 14" x 16 ply composites by resin transfer molding (RTM) from all NASA supplied resins

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

Chemistry of PETI-298



Nitrogen NMP
 35-50% solids

Amide Acid Oligomer

Toluene Reflux

Imide Oligomer (Soluble)

Calculated Mn 750 g/mole = PETI-298

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

Comparison of PETI Oligomers Prepared From 1,3,3 and 1,3,4 - APB

APB Diamine	Calculated Mn, g/mole	Glass Transition Temp., °C Initial	Cured*	Melt Viscosity @ 280°C, poise
1,3,3	750	132	258	1-6
1,3,3	1250	151	244	5-15
1,3,4	750	139	298	6-13
1,3,4	1250	165	285	10,000**

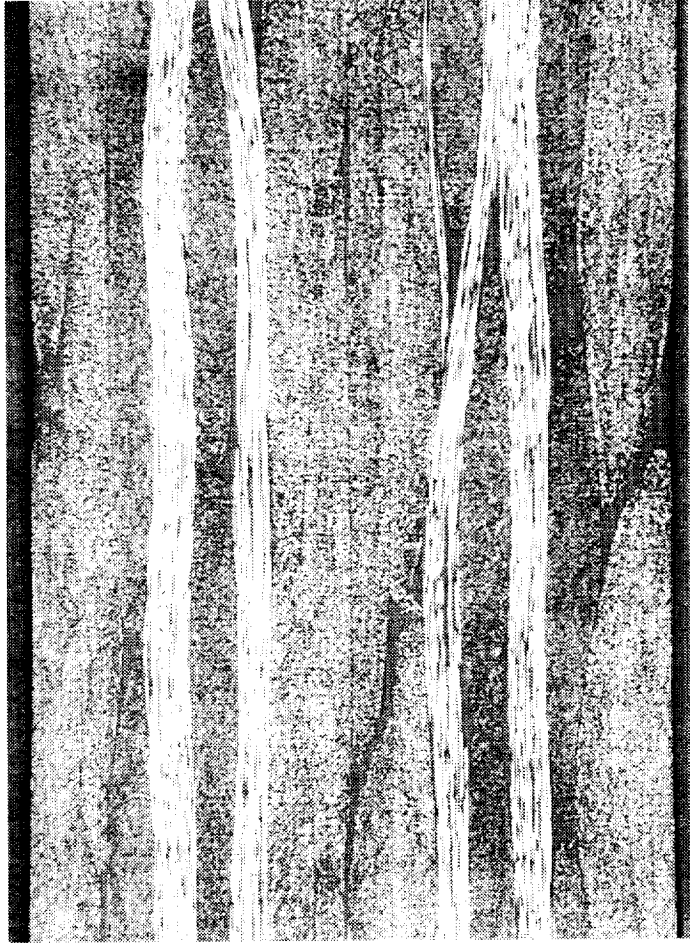
* Cured 1 hour at 371°C

**Viscosity dropped to
~30 poise at 325°C

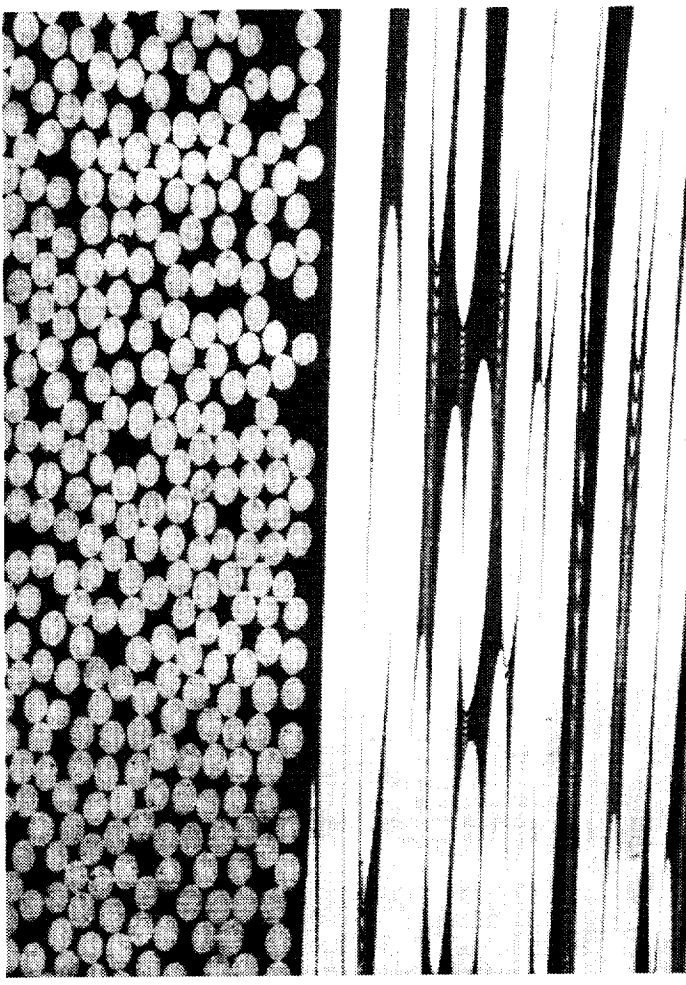
3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

Photomicrographs of PETI-298 Laminates Fabricated Via RTM



25 x Magnification

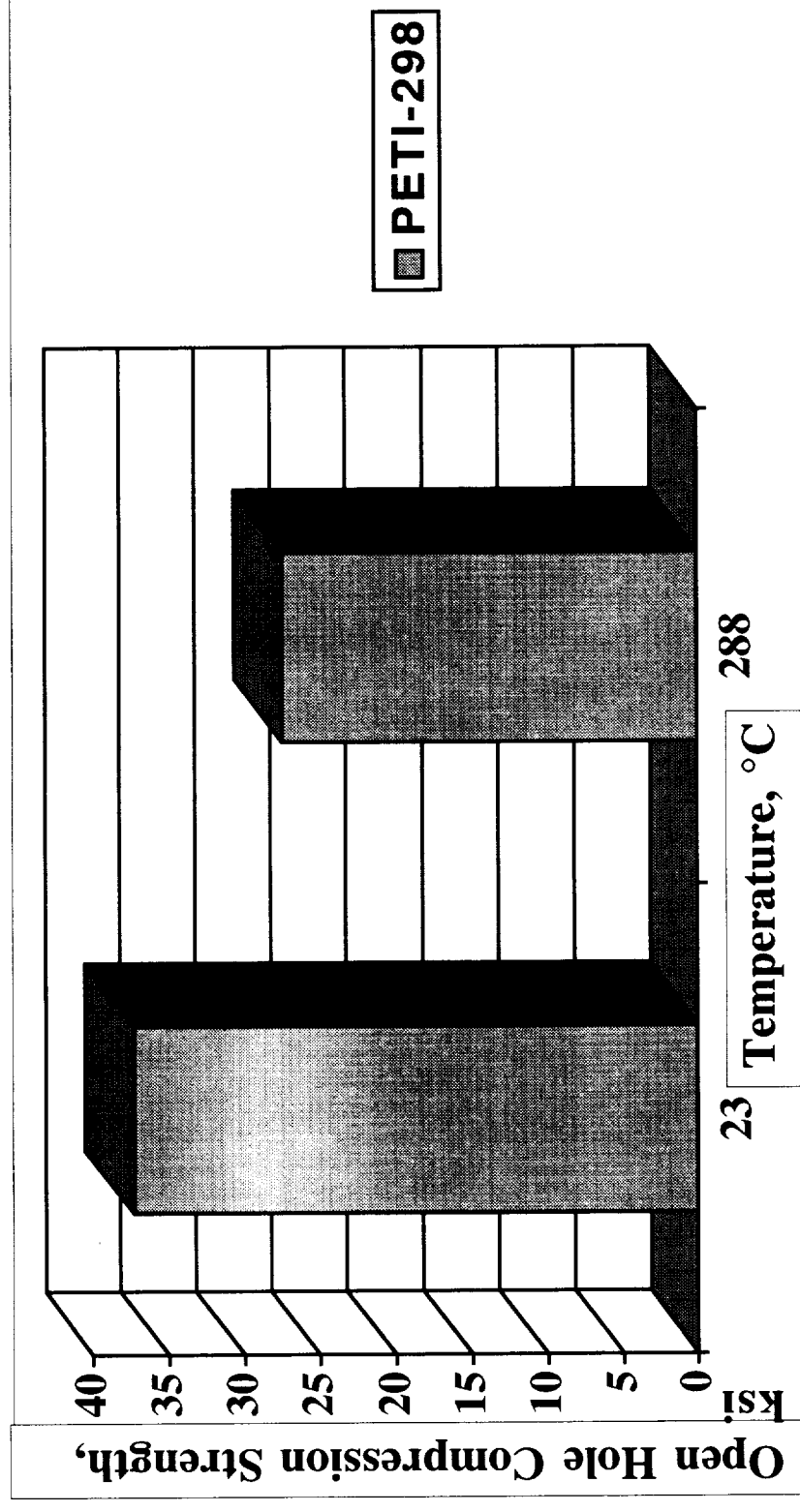


400 x Magnification

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

Mechanical Properties of AS-4/PETI-298 Fabric Composites Fabricated Via Resin Transfer Molding (8 ply)



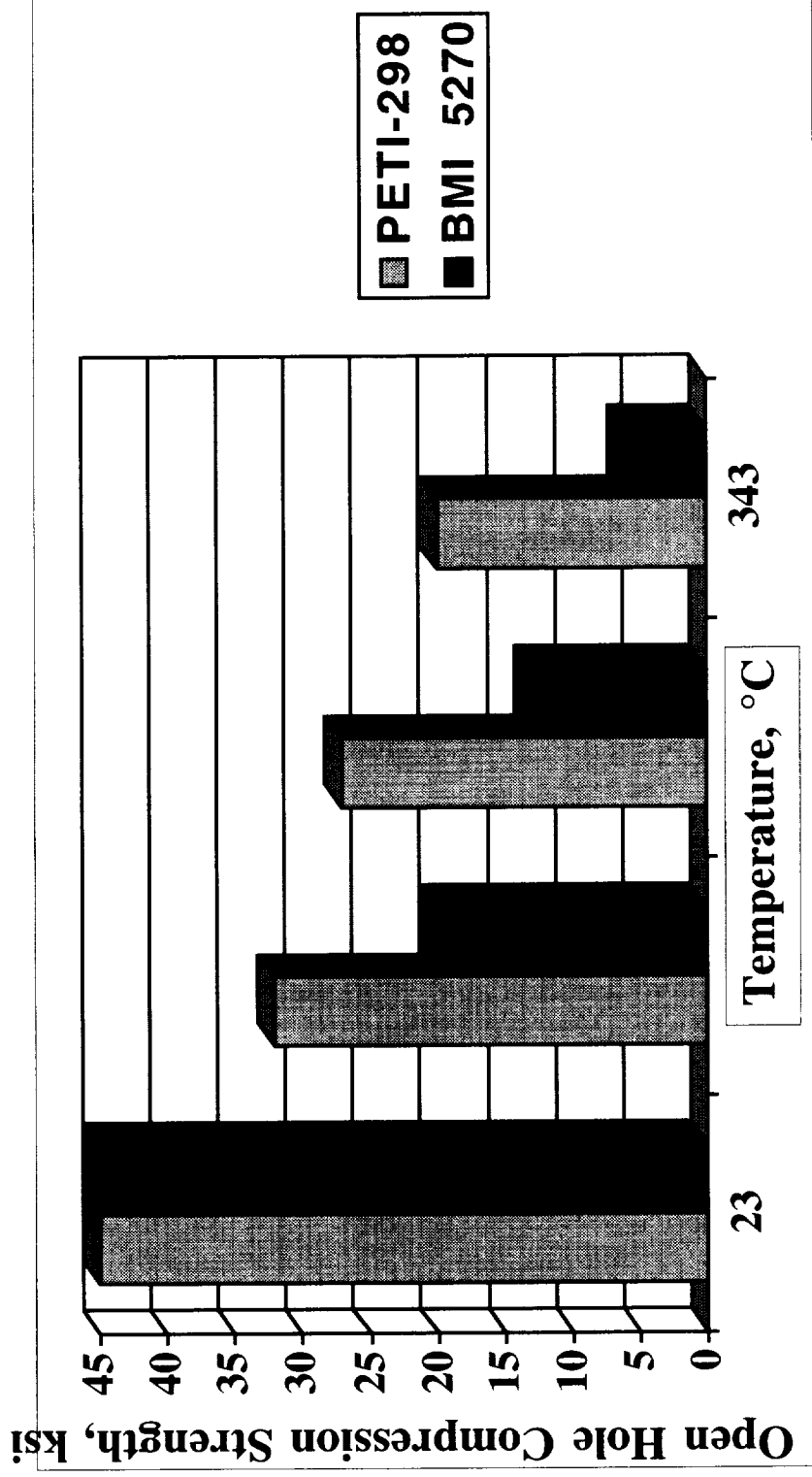
PETI-298 cured 1 hr @ 370°C, T_g = 302°C (8 ply AS-4 fabric)

Un-notched Compression Strength at 23°C = 60 ksi

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

Mechanical Properties of IM-7 PETI-298 Stitched Composites Fabricated Via Resin Infusion (36 ply)



PETI-298 cured 1 hr @ 370°C, postcured at 370°C, T_g = 338°C (Panel 36 ply x 22" x 22", stitched)

BMI 5270 cured 4 hr @ 190°C, postcured at 232 and 260°C, T_g = 299°C

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

- ♦ Resins for transfer molding or infusion processing
 - POC - Paul M. Hergenrother, NASA LaRC
- ♦ Nonautoclave processable adhesives
 - POC - Brian J. Jensen, NASA LaRC
- ♦ Automated Tape Placement Device with e-beam cure
 - POC - Harry L. Belvin, NASA LaRC

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

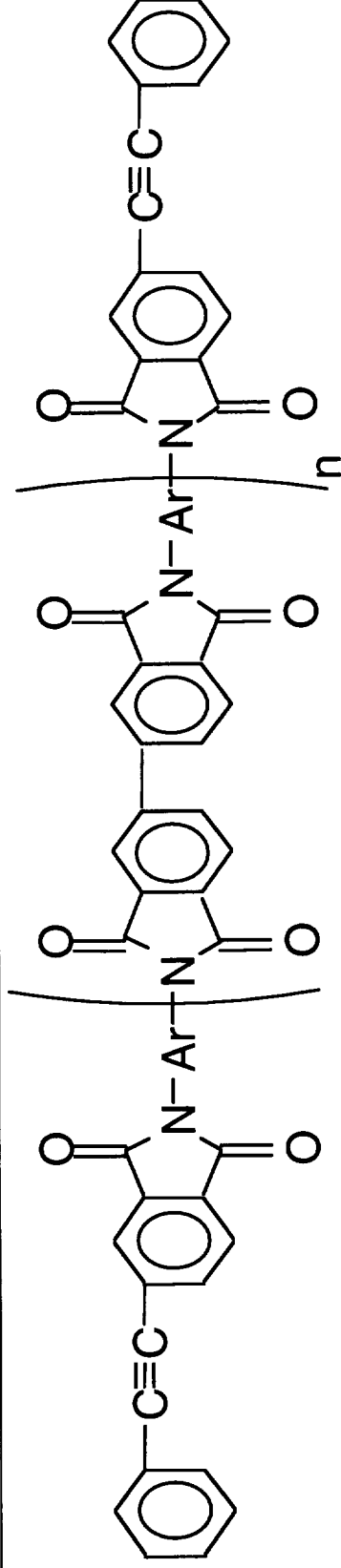
Accomplishments, LaRC PETI-8

- ♦ Developed and supplied to Cytec Fiberite several non-autoclave processable adhesives.
- ♦ LaRC PETI-8 is a phenylethynyl terminated polyimide adhesive which has low melt viscosity and excellent melt stability at temperatures below 300°C, allowing the production of excellent adhesive bonds under vacuum bag pressure, without the need for external pressure normally supplied by an autoclave. Heating at 316°C for 8 hours provides excellent titanium to titanium tensile shear strengths from 75°F to at least 350°F and excellent flatwise tensile strengths at 75°F.
- ♦ Plan to continue work on adhesives which do not require an autoclave for processing. Concentrate on vacuum bag / oven processing, hot melt adhesives and the use of e-beam radiation to cure advanced adhesives. Optimize the properties of LaRC PETI-8 by studying various formulations of the adhesive tape and various cure conditions.

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

LaRC PETI-8



Titanium to Titanium Tensile Shear Strengths

Required

5000 psi at 75° F

3500 psi at 350° F

Achieved

7400 psi

6200 psi

Flatwise Tensile Strength (Composite Skins over Titanium core)

Required

1000 psi at 75° F

Achieved

1370 psi

Bonding Conditions:

Vacuum Bag Only Pressure, 316°C, 8 hour hold, 5V CAA surface treatment

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

Cytec Fiberite Results for PETI-8 Bonding

Evaluated 550°F, 575°F and 600°F cycles from 4-12 hours under vacuum bag only pressure for several different formulations. Shown are results for 600°F, 4 hour cycle.

PETI-8 Tensile Shear Strength	<u>75°F</u>	<u>350°F</u>
Titanium substrate, CAA Anodized	7000 psi (min.)	5000 psi (min.)
PETI-5 composite substrate (interlaminar failure at both test temperatures)	5500 psi	4500 psi

PETI-8 Flatwise Tensile Strength	<u>75°F</u>
2024 Al face sheets, FPL etched, 3/16" Ti core	1800 psi

Cytec currently preparing two 2' x 2' PETI-5 composite panels to be bonded together as a wide area specimen.

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

- ◆ Resins for transfer molding or infusion processing
 - POC - Paul M. Hergenrother, NASA LaRC
- ◆ Nonautoclave processable adhesives
 - POC - Brian J. Jensen, NASA LaRC
- ◆ Automated Tape Placement Device with e-beam cure
 - POC - Harry L. Belvin, NASA LaRC

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials

Accomplishments, ATP with E-Beam Cure

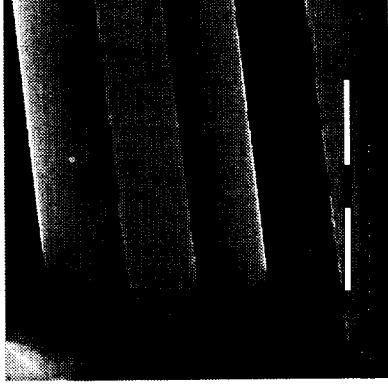
- ♦ GRC has Cooperative Agreement with Kent State University to study e-beam irradiation of polyimide thin films. (Shows little effect on mechanical properties or Tg)
- ♦ GRC has Cooperative Agreement with University of Delaware to study new e-beam curable resins. (Extent of cure dependent on molecular mobility)
- ♦ GRC in-house e-beam curable resin development. (Diels-Alder trapping of quinodimethane intermediates formed under radiation)
- ♦ LaRC and Boeing developing a tape laying machine with e-beam cure-on-the-fly processing. Undergoing acceptance testing at Boeing and will be shipped to LaRC when facilities are ready.

3rd Gen Airframe/TPS:

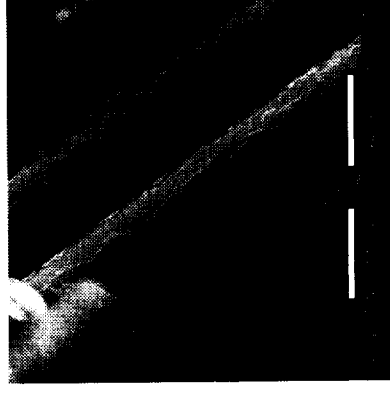
Int. Thermal Structures and Materials

♦ Products/ Benefits/Payoff:

- Validate the cause of low performance in E-beam cured graphite/epoxy composites and investigate methods for improving their performance through the use of novel sizings or resin additions.
- The goals are to:
 - Positively identify the deficiencies causing reduced properties in E-beam cured composites
 - Identify and demonstrate the best method for performance improvement
- Improved performance of E- beam composites will enable out-of-autoclave fabrication of large cryo tanks. Higher performance of these materials directly reduces RLV vehicle weight.



E-Beam Cured Cat-B



Thermally Cured 8552

3rd Gen Airframe/TPS:

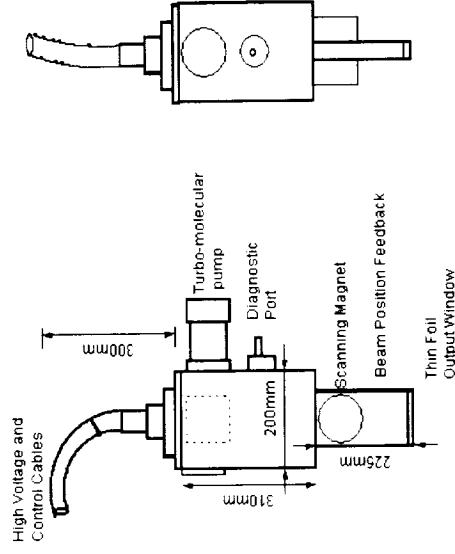
Int. Thermal Structures and Materials

E-beam Gun Head from Electron Solutions, Inc. E-beam Gun Head from Electron Solutions, Inc. Boeing Tape Laying Gantry

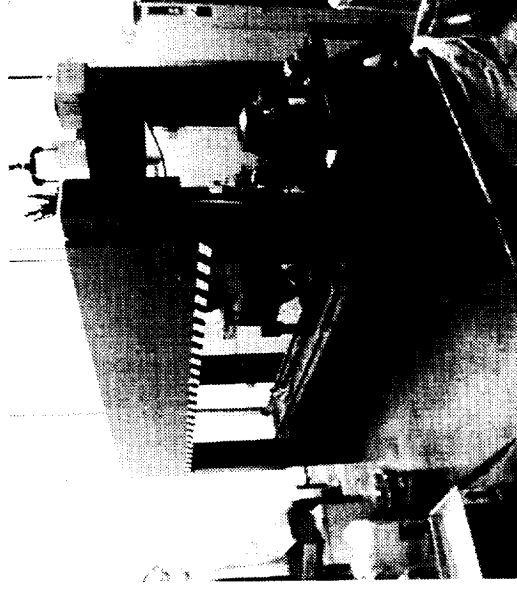
This task will design, fabricate and deliver a tape laying device capable of laying E-beam "cure-on-the-fly" (COTF) prepreg for material evaluations.

• Products/ Benefits/Payoff:

COTF E- beam curing will enable out-of-autoclave fabrication of RLV cryo tanks which will substantially reduce overall vehicle weight.



E-beam Gun Head from
Electron Solutions, Inc.



Boeing Tape Laying Gantry

3rd Gen Airframe/TPS:

Int. Thermal Structures and Materials